

## SCIENCE FOR CERAMIC PRODUCTION

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### INVESTIGATION OF THE STRUCTURE AND PHASE COMPOSITION OF ALUMINUM OXIDE POWDERS OBTAINED BY CHEMICAL DISPERSION OF AN ALUMINUM ALLOY WITH DIFFERENT MAGNESIUM CONTENT

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The results of x-ray phase and petrographic analyses and scanning electron microscopy of aluminum hydroxide powders, obtained by chemical dispersion of aluminum-magnesium alloys (with Mg mass fraction 0.6, 6 and 12% in the alloys), as well as powders obtained by calcination of these hydroxides are presented. A mechanism is proposed for structure formation in powders at different stages of the technological process used to make the aluminum hydroxide: the product of the chemical dispersion of alloy is a powder after heat treatment in air (1400°C, 1 h).

**Key words:** gibbsite, bayerite, chemical dispersion, aluminum-magnesium alloy, heat treatment, structure, aluminomagnesia spinel.

The properties of a ceramic material are determined by the structure of the material comprising the experimental sample irrespective of the method used to obtain this structure. The term 'structure' is currently understood to mean the spatial organization of the substance comprising the material, characterized by all morphometric, geometric and energy indices and determined by the composition, quantitative ratio and interaction of the components of the material [1]. For this reason there is special interest in finding ways to control the properties of the materials by means of structural changes. In physical-chemical analysis this problem was formulated as a problem of dispersity – composition – structure – property [2]. The first part of this dependence dispersity – composition – structure pertains to the technology used to fabricate the material, i.e., the paths for obtaining a given structure. The second part of this chain structure – property determines the user quality of the product, i.e., its serviceability. For this reason the structure – property link is one of the main problems of ceramic materials science.

The development of new material based on aluminum oxide and articles based on it are indissolubly related with the development of new methods of obtaining the initial

powders. In addition, at the stage where the powders themselves are obtained it is necessary to obtain powders with particles having prescribed structure, size and shape as well phase composition. This will make it possible to produce a ceramic with a definite structure and the requisite user properties.

The aim of the present work is to study the structure and phase composition of the powders obtained by chemical dispersion of an aluminum alloy with different magnesium content.

#### PRODUCTION OF THE EXPERIMENTAL SAMPLES

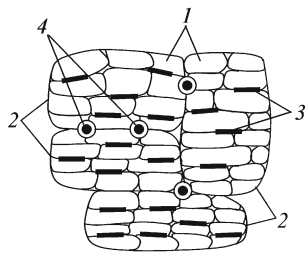
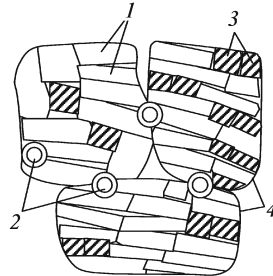
Aluminum hydroxide powders were obtained by chemical dispersion of an Al–Mg alloy with different magnesium mass fractions (0.6, 6 and 12%) in a 20% solution of sodium hydroxide [3]. The precipitate obtained was filtered out of the mother liquor and repeatedly washed with distilled water by filtration in a vacuum. One liter of water was consumed in each wash cycle. Twelve wash cycles were conducted. The precipitate was dried at temperature 60°C (in air). A free-flowing powder consisting of mixed hydroxides was obtained after drying. The precipitate was subjected to heat-treatment for 1 h in air at temperature 1400°C. At this temperature the mixed hydroxides comprising the precipitate

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**TABLE 1.** Structure and Phase Composition of the Powder Obtained by Chemical Dispersion of the Alloy Al–Mg (Mg mass fraction 0.6%)

| Mg mass fraction<br>in the initial alloy, % | Structure and phase composition of the powder   |   |
|---|---|---|
|   | P <sub>11</sub> after drying* at 60°C   | P <sub>12</sub> after heat-treatment* at 1400°C, 1 h  |
| 0.6   | Predominant phase Al(OH) <sub>3</sub> — gibbsite  | Predominant phase α-Al <sub>2</sub> O <sub>3</sub>  |
|   |  <p>1) basal plates — submicron Al(OH)<sub>3</sub> crystals;<br/>2) agglomerates comprised of basal plates (length 4 – 8 μm, width 0.1 – 6 μm);<br/>3) phase based on the complex hydroxide NaMgAlOH — submicron crystals;<br/>4) inclusions of the alloy in a metal hydroxide shell</p> |  <p>1) short-prismatic α-Al<sub>2</sub>O<sub>3</sub> crystals (length 2 – 3 μm, width 1.5 – 1.7 μm) — 75 – 80%;<br/>2) hollow rounded granules of α-Al<sub>2</sub>O<sub>3</sub> (5 – 15 μm);<br/>3) cubic Al<sub>2</sub>MgO<sub>4</sub> crystals (0.5 – 1.0 μm) — 2 – 3% above 100%;<br/>4) agglomerates</p> |

\* Drying and heat treatment were conducted in air.

transformed into the corresponding oxides. The structure and phase composition of the powders were studied by means of petrographic and crystal-optic analyses.

## ANALYSIS OF THE STRUCTURE AND PHASE COMPOSITION OF THE POWDERS

### 1. Al–Mg powder with Mg mass fraction 0.6%

The amount of the alloy Al–Mg present in the powder P<sub>11</sub> after drying is quite significant (to 25 vol.%). Residues of this alloy are coated with a metal hydroxide shell comprised of submicron gibbsite crystals. After heat treatment at 1400°C these shells transform into α-Al<sub>2</sub>O<sub>3</sub> in the product P<sub>12</sub>. The residual inclusions of the alloy go into the gas phase, leave the cavities and condense in the interior volume of the material. Cavities form at the locations of granules, where the residues of the alloy were located. Together with the predominant phase α-Al<sub>2</sub>O<sub>3</sub> (2 – 3 μm crystals) formed from gibbsite, spinel (0.5 – 1 μm crystals) also forms from the complex hydroxide NaMgAlOH during heat treatment.

The results of the petrographic analysis and the structure of the powders before and after calcination are presented in Table 1.

### 2. Al–Mg powders with Mg mass fraction 6%

The amount of the initial Al–Mg alloy present in the P<sub>21</sub> powder after drying is larger (about 30 vol.%) than in the powder P<sub>11</sub>. Probably, this is associated with the particulars of powder production by chemical dispersion of the corresponding alloys. The residues of this alloy were coated with

a metal hydroxide shell, which is represented by submicron crystals of bayerite. After heat treatment at 1400°C these shells in the P<sub>22</sub> powder transform into α-Al<sub>2</sub>O<sub>3</sub>.

Crystals of a Mg-containing phase in the form of the solid solution Al<sub>2</sub>O<sub>3</sub>–MgO, formed from a solid substitution solution of Mg<sup>2+</sup> in Al(OH)<sub>3</sub> present in the precipitate (in the powder product P<sub>21</sub>), lie along the boundaries of the α-Al<sub>2</sub>O<sub>3</sub> crystals. The residual inclusions of the alloy, just as in the preceding case, go into the gas phase, leave the cavities and condense in the interior volume of the material with cavities forming in the interior volume of the powder. Crystals of an Mg-containing component in the form of the solid solution Al<sub>2</sub>O<sub>3</sub>–MgO, formed from the solid substitution solution of Mg<sup>2+</sup> in Al(OH)<sub>3</sub>, lie along the boundaries of α-Al<sub>2</sub>O<sub>3</sub> crystals.

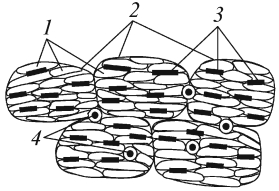
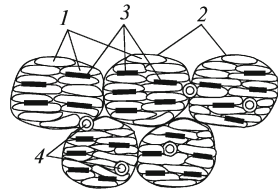
The results of petrographic analysis and the structure of the powders before and after calcination are presented in Table 2.

### 3. Powders of the alloy Al–Mg with Mg mass fraction 12%

The powders P<sub>31</sub> and P<sub>32</sub> obtained from this alloy exhibit characteristic differences from the powders obtained from the alloy Al–Mg with Mg mass fraction 0.6 and 6%.

The dried powder P<sub>31</sub> consists predominantly of rounded granules with a layered structure. In contrast to the powders P<sub>11</sub> and P<sub>21</sub>, the spherical nucleus consisting of an aluminum alloy (10 – 12 μm) is covered by alternating shells consisting of amorphous boehmite, bayerite and an amorphous hydrate phase (Al<sub>2</sub>O<sub>3</sub>–MgO) · nH<sub>2</sub>O atop which lie submicron particles of bayerite.

**TABLE 2.** Structure and Phase Composition of the Powder Obtained by Chemical Dispersion of the Alloy Al–Mg (Mg mass fraction 6%)

| Mg mass fraction<br>in the initial alloy, % | Structure and phase composition of the powder   |   |
|---|---|---|
|   | P <sub>21</sub> after drying* at 60°C   | P <sub>22</sub> after heat-treatment* at 1400°C, 1 h  |
| 6.0   | Predominant phase Al(OH) <sub>3</sub> – bayerite<br><br>1) lamellar crystals of bayerite (0.5 – 0.7 μm);<br>2) agglomerates – porous spherulites (15 – 20 μm);<br>3) solid substitution solution of Mg <sup>2+</sup> in Al(OH) <sub>3</sub> ;<br>4) inclusions of the alloy in a metal hydroxide shell | Predominant phase α-Al <sub>2</sub> O <sub>3</sub><br><br>1) lamellar α-Al <sub>2</sub> O <sub>3</sub> crystals (0.5 – 1 μm);<br>2) agglomerates (spherulites) (50 – 80 μm);<br>3) solid solution of Al <sub>2</sub> O <sub>3</sub> –MgO (crystal size 0.5 – 1.0 μm);<br>4) hollow rounded granules α-Al <sub>2</sub> O <sub>3</sub> |

\* Drying and heat treatment were conducted in air.

Two predominant phases — α-Al<sub>2</sub>O<sub>3</sub> and spinel — are recorded in the structure of the P<sub>32</sub> powder after heat treatment. They form mainly from bayerite and shells of an amorphous hydrate phase (Al<sub>2</sub>O<sub>3</sub>–MgO) · nH<sub>2</sub>O, which crystallize into α-Al<sub>2</sub>O<sub>3</sub> and spinel. During heat treatment the shell of the amorphous hydrate phase breaks down and detaches from the bayerite shell because of the difference of their CLTE. The alloy inclusion (nucleus) is found to be closed in the α-Al<sub>2</sub>O<sub>3</sub> shell, formed as a result of the diffusion splicing of layers of amorphous boehmite and fine-crystalline bayerite. In this case, the initial stage in the formation of an internal cavity, owing to incomplete evaporation of alloy par-

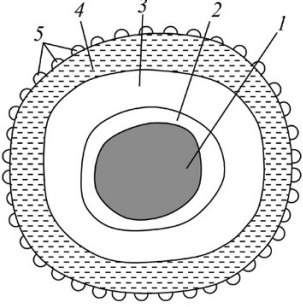
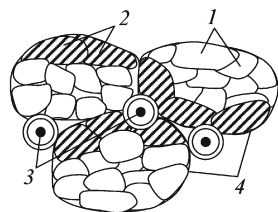
ticles and transport of the vapor molecules through the aluminum oxide shell followed by their condensation in the form of crystals of α-Al<sub>2</sub>O<sub>3</sub> and spinel, is observed.

The results of the petrographic analysis and the structure of the P<sub>31</sub> and P<sub>32</sub> powders before and after calcination are presented in Table 3.

## CONCLUSIONS

The structure and phase composition of aluminum hydroxide powder obtained by chemical dispersion of the alloy Al–Mg (0.6, 6, and 12%) and powders obtained during heat treatment of these hydroxides at 1400°C were investigated.

**TABLE 3.** Structure and Phase Composition of the Powder Obtained by Chemical Dispersion of the Alloy Al–Mg (Mg mass fraction 12%)

| Mg mass fraction<br>in the initial alloy, % | Structure and phase composition of the powder  |  |
|---|--|--|
|   | P <sub>31</sub> after drying* at 60°C  | P <sub>32</sub> after heat-treatment* at 1400°C, 1 h   |
| 12  | Predominant phase Al(OH) <sub>3</sub> – bayerite<br><br>1) nucleus — aluminum alloy (10 – 12 μm);<br>2) amorphous boehmite (1.5 – 6 μm layer);<br>3) bayerite (20 – 60 μm layer);<br>4) amorphous hydrate phase (Al <sub>2</sub> O <sub>3</sub> –MgO) · nH <sub>2</sub> O;<br>5) submicron and micron particles of bayerite | Predominant phases α-Al <sub>2</sub> O <sub>3</sub> and Al <sub>2</sub> MgO <sub>4</sub><br><br>1) α-Al <sub>2</sub> O <sub>3</sub> crystals (0.8 – 1 μm);<br>2) Al <sub>2</sub> MgO <sub>4</sub> crystals (1 – 3.5 μm);<br>3) inclusions of an Al alloy (2 μm), covered with an α-Al <sub>2</sub> O <sub>3</sub> shell (initial stage in the formation of a cavity);<br>4) agglomerates (12 – 15 μm) |

\* Drying and heat treatment were conducted in air.

It was shown that the phases predominating in the P<sub>32</sub> powder ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>MgO<sub>4</sub>) are formed in P<sub>31</sub> powder during heat treatment.

It was found that hollow rounded granules with an aluminum oxide shell form in the powders P<sub>12</sub>, P<sub>22</sub> and P<sub>32</sub>. These cavities probably form by evaporation – condensation.

These powders can be used for the production of articles from corundum ceramic.

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